

PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY


(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

REC'D 11 OCT 2005

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Applicant's or agent's file reference 58745WO003		FOR FURTHER ACTION		See Form PCT/PEA416
International application No. PCT/EP2004/007745		International filing date (day/month/year) 14.07.2004	Priority date (day/month/year) 14.07.2003	
International Patent Classification (IPC) or national classification and IPC A61K6/00				
Applicant 3M ESPE AG et al				
<p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p>a. <input checked="" type="checkbox"/> sent to the applicant and to the International Bureau a total of 5 sheets, as follows:</p> <p><input checked="" type="checkbox"/> sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).</p> <p><input type="checkbox"/> sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.</p> <p>b. <input type="checkbox"/> (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).</p>				
<p>4. This report contains indications relating to the following items:</p> <p><input checked="" type="checkbox"/> Box No. I Basis of the opinion</p> <p><input type="checkbox"/> Box No. II Priority</p> <p><input type="checkbox"/> Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p><input type="checkbox"/> Box No. IV Lack of unity of invention</p> <p><input checked="" type="checkbox"/> Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p><input type="checkbox"/> Box No. VI Certain documents cited</p> <p><input type="checkbox"/> Box No. VII Certain defects in the international application</p> <p><input type="checkbox"/> Box No. VIII Certain observations on the international application</p>				
Date of submission of the demand 15.03.2005		Date of completion of this report 11.10.2005		
Name and mailing address of the international preliminary examining authority:  European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016		Authorized Officer Böhm, I Telephone No. +31 70 340-1050		



**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/EP2004/007745

Box No. I Basis of the report

1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ This report is based on translations from the original language into the following language , which is the language of a translation furnished for the purposes of:
- ☐ international search (under Rules 12.3 and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4)
 - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)
2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

Description, Pages

1-21 as originally filed

Claims, Numbers

1-16 received on 19.09.2005 with letter of 19.09.2005

- ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing
3. ☐ The amendments have resulted in the cancellation of:
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
 - ☐ any table(s) related to sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
 - ☐ any table(s) related to sequence listing (*specify*):

* If item 4 applies, some or all of these sheets may be marked "superseded."

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/EP2004/007745

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	1-16
	No: Claims	
Inventive step (IS)	Yes: Claims	1-16
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-16
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Re Item V.

The following documents have been referred to in this communication:

D1 : EP 0 842 651 A (MUEHLBAUER ERNST KG) 20 May 1998 (1998-05-20)

D2 : EP 0 712 622 A (TOKUYAMA CORP) 22 May 1996 (1996-05-22)

I) Novelty

D1 (EP 0842651 A) discloses a primer composition for dental composite fillers. The primer composition is an adhesive based on phosphoric acid and 10-90 wt.-% of phosphoric acid monoester and phosphoric acid diester. (see p.3, line 25-57)

This composition combines both steps, the etching-step and applying the primer. (see p.4, lines 39-41)

The composition contains 20-35 wt.-% of phosphoric acid monoester and 25-45 wt.-% of phosphoric acid diester. (see cl. 6) The composition contains furthermore a polymerisation initiator. (see cl.12)

Additionally the composition comprises 1-15 wt.-% of a phosphoric acid triester and a diphosphate. (see p.4, lines 1-20)

The phosphoric acid groups containing (meth)acrylic acid groups, these groups are polymerising and forming a polymerized film on the surface of the tooth.

D1 does not disclose a composition comprising component (b), that is one or a mixture of phosphoric acid esters bearing substituents, wherein one of the substituents has two or more ethylenically unsaturated moieties on the phosphorus atom; in contrast to that, the unsaturated moieties of components (b) of D1 are located in different substituents (R1 and R2).

Therefore the subject-matter of claims 1-16 is novel of D1. (Article 33(1) and (2) PCT)

II) Inventive step

D1 represents the **closest prior art** and discloses a primer composition for dental composite fillers. The primer composition is an adhesive based on phosphoric acid and 10-90 wt.-% of phosphoric acid monoester and phosphoric acid diester.

The present invention **differs** from D1 in that D1 fails to disclose the specific combination of components (a) and (b).

The **problem** to be solved is to provide a dental composition showing good adhesion to a tooth surface and having low water uptake.

The inventive composition of claim 1 **solves** the problem posed having the technical effect of decrease in polarity upon curing as well as good adhesion properties.

The decrease in polarity is combined with a lower water uptake and thus reduced swelling.

The skilled person has no motivation and suggestions from D1 to arrive at the dental composition claimed.

Therefore the subject-matter of claims 1-16 involves an **inventive step**. (see Article 33(1) and (3) PCT)

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Claims:

5 1. A dental composition, comprising

a) one or a mixture of phosphoric acid esters bearing substituents with one ethylenically unsaturated moiety on the phosphorus atom,

b) one or a mixture of phosphoric acid esters bearing substituents ^[-] with two or more ethylenically unsaturated moieties on the phosphorus atom;

c) initiators, ^[, wherein one of the substituents has]

d) optionally stabilizers,

e) unsaturated monomers

f) optionally unsaturated polymers and/or prepolymers

g) optionally a solvent,

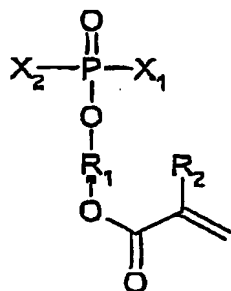
h) optionally a fluoride release agent,

i) optionally a non reactive inorganic filler,

j) optionally a photobleachable colorant,

wherein $\text{C}=\text{C}$

2. The dental composition according to claim 1, wherein component (a) is represented by formula (I)



(I)

The term "on the phosphorus atom" means that the ethylenically unsaturated moiety/moieties is/are present in one of the substituents bonded to the phosphorus atom.

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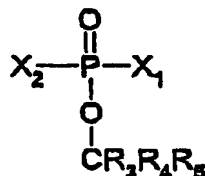
wherein R_1 = (i) alkylene having 1 to 4 C atoms, (ii) or a bivalent organic group having 1 to 4 carbon atoms composed of two or more hydrocarbon residues bonded to one another by one or more ether or thioether linkages, (iii) or aryl, each optionally substituted with OH;

5 wherein R_2 = H, CH_3 ;

wherein X_1 = OH or halogen; and

wherein X_2 = X_1 or $-\text{O}-R_1-\text{OOC}-\text{CR}_2=\text{CH}_2$,

and component (b) is represented by formula (II),



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(II)

wherein R_3 , R_4 , and R_5 = (i) H, (ii) linear or branched alkyl having 1 to 4 carbon atoms, optionally substituted with OH, (iii) aryl, optionally substituted with OH, (iv) organic group having 5 to 15 carbon atoms composed of 2 to 6 saturated or ethylenically unsaturated hydrocarbon residues bonded to one another by one or more ether, thioether, ester, thioester, thiocarbonyl, amide, urethane, carbonyl and/or sulfonyl linkages, each optionally substituted with OH,

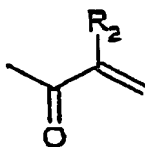
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wherein at least 2 of the groups R_3 , R_4 , and R_5 comprise at least 1 group according to formula (III)

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or

at least 1 of the groups R_3 , R_4 , and R_5 comprises at least 2 groups according to formula (III)



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(III)

and wherein $X_2 = X_1$ or $-O-CR_3R_4R_5$ or $-O-R_1-OOC-CR_2=CH_2$.

- 5 3. The dental composition according to anyone of the preceding claims, wherein component (b) is present in an amount of about 1 to about 500 parts by weight based on about 100 parts by weight of component (a).
- 10 4. The dental composition according to anyone of the preceding claims, wherein the total amount of components (a) and (b) in the composition is about 10 to about 90 parts by weight.
- 15 5. The dental composition according to anyone of the preceding claims, wherein the polymer and/or prepolymer of component (f) is present in an amount of about 0 to about 30 parts by weight.
6. The dental composition according to claim 5, wherein the prepolymer does not contain any hydroxy, acidic or ionic groups.
- 20 7. The dental composition according to claim 5 or 6, wherein the prepolymer has an Mw in the range of about 600 to about 20000.
- 25 8. The dental composition according to anyone of the preceding claims having a contact angle versus deionized water of more than about 15° , if the composition is cured in the presence of air, and of more than about 50° , if the composition is cured in the absence of air.

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9. The dental composition according to anyone of the preceding claims having an adhesion to enamel and/or dentin in the range of about 2 to about 15 MPa.

5 10. The dental composition according to anyone of the preceding claims having a water uptake of less than about 5 % by weight with respect to the cured composition measured after having immersed the composition for 5 h in water of 37°C.

10 11. The dental composition according to claim 10 having an enamel adhesion of at least about 5 MPa.

15 12. The dental composition according to anyone of the preceding claims, wherein component (a) is selected from 2-methacryloyloxyethyl phosphate, 2-methacryloyloxypropyl phosphate, 3-methacryloyloxypropyl phosphate, 2-methacryloyloxybutyl phosphate, 3-methacryloyloxybutyl phosphate, 4-methacryloyloxybutyl phosphate, 5-methacryloyloxy-3-oxa-pentyl phosphate, bis(2-methacryloyloxyethyl) phosphate, bis(2-methacryloyloxypropyl) phosphate, bis(3-methacryloyloxypropyl) phosphate, bis(2-methacryloyloxybutyl) phosphate, bis(3-methacryloyloxybutyl) phosphate, bis(4-methacryloyloxybutyl) phosphate, bis(5-methacryloyloxy-3-oxa-pentyl) phosphate, phosphate, or mixtures thereof.

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25 13. The dental composition according to anyone of the preceding claims, wherein component (b) is selected from glycerol-1,3-dimethacrylate-2-phosphate, glycerol-1,2-dimethacrylate-3-phosphate, bis(glycerol-1,3-dimethacrylate) phosphate, bis(glycerol-1,2-dimethacrylate) phosphate, (glycerol-1,2-dimethacrylate)(glycerol-1,3-dimethacrylate) phosphate, (trimethylolpropane dimethacrylate) phosphate, bis(trimethylolpropane dimethacrylate) phosphate, (trimethylolethane dimethacrylate) phosphate, bis(trimethylolethane

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dimethacrylate) phosphate, pentaerythritol trimethacrylate phosphate or mixtures thereof.

- 5 14. Kit of parts, comprising in one part components (a), (b), (c), (d), (e), (f), (h), (i), (j) and in another part components (c), (d), (e), (f), (g) as described in anyone of the preceding claims.
- 10 15. Use of a dental composition according to anyone of claims 1 to 13 or the kit according to claim 14 for the production of a material to be used for the adhesive securing of dental filling materials based on (meth)acrylates and/or epoxies, as self-adhesive dental pit and fissure sealants, and/or as self-adhesive dental desensitizers.
- 15 16. Method for preparing a composition according to anyone of claims 1 to 14 comprising the step of mixing the components.